

Utilization of Metakaolin-Based Geopolymers for Stabilization of Sulfate-Rich Expansive Soils

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ABSTRACT

The durability and performance of pavements and other lightweight structures constructed over expansive soils are persistent causes of concern due to moisture-induced volumetric fluctuations. Typically, traditional calcium (Ca)-based stabilizers, especially lime, effectively mitigate the problems associated with such problematic soils by reducing their swell-shrink potential and improving their strength properties. However, the treatment of sulfate-rich expansive soils using the Ca-based stabilizers results in detrimental heave due to the formation of a highly deleterious mineral, ettringite. This research study was performed to investigate the efficacy of utilizing an eco-friendly metakaolin-based geopolymer (MKG) stabilizer for treating sulfate-rich expansive soils. A comparative study was conducted by performing engineering tests such as free swell tests and unconfined compressive strength (UCS) tests on untreated, lime-treated, and geopolymer-treated soils for different curing periods. The effects of geopolymer dosage, curing period, and moisture intrusion on swelling characteristics and strength properties of treated soils were investigated. In addition, microstructural analyses were performed to study the changes in the soil after chemical treatment with geopolymer and lime. The results of engineering tests and microstructural studies indicate that geopolymer can effectively stabilize sulfate-rich expansive soils and could be used as a sustainable and eco-friendly alternative to traditional Ca-based stabilizers.

INTRODUCTION AND BACKGROUND

Expansive soils are prevalent in different parts of the world and exhibit excessive swelling and shrinking characteristics with moisture fluctuations (Hoyos et al. 2006, Puppala et al. 2006, Biswas et al. 2021c, 2021b). Traditional Ca-based stabilizers, especially lime, have been used for decades to mitigate moisture-induced volumetric changes and enhance engineering properties of expansive soils (Thompson 1968, Du et al. 2016, Little 2000). Lime treatment results in immediate modification of the treated soil by cation exchange and flocculation agglomeration

reactions, which reduce soil plasticity, swelling and shrinkage potential, change the texture, and make it workable (Mitchell and Dermatas 1992, Little and Nair 2009, Biswas et al. 2021a). Furthermore, lime treatment results in long-term pozzolanic reaction and forms cementitious phases like calcium silicate hydrate (C-S-H) and calcium aluminate hydrate (C-A-H), which enhances the strength and stiffness properties of the treated soil matrix (Dash and Hussain 2012, Chakraborty and Nair 2020). Even though lime is often found to be very effective in stabilizing expansive soils, the presence of soluble sulfate salts like gypsum and anhydrite makes lime treatment ineffective and often counterproductive due to the formation of a highly expansive mineral, ettringite (Mitchell 1986, Kota et al. 1996).

Ettringite, a tricalcium-alumino-sulfate-hydrate compound, is formed when the alumina released from clay minerals in a high pH environment ($\text{pH} > 10.5$) reacts with the calcium and sulfate ions in the presence of water (Hunter 1988, Wild et al. 1999). Stoichiometrically, ettringite formation can result in a volumetric expansion of 137% (Little et al. 2010, Talluri et al. 2020). This expansive nature of ettringite has a detrimental impact on civil infrastructures. Hence, traditional lime treatment is not recommended for expansive soils with soluble sulfate levels greater than 3,000 ppm (TxDOT 2019). Instead, modified lime treatment techniques including pre-compaction mellowing, double lime application, and utilization of co-additives like low-calcium fly ash, ground granulated blast furnace slags, crystalline silica-rich admixtures have been used and recommended for stabilizing sulfate soils (Wild et al. 1999, Chakraborty et al. 2020, Khadka et al. 2020). Even though these methods have shown promising results, most of these methods increase the construction time to account for the mellowing time delays or require long curing time for the treatment to be effective before moisture exposure (Chakraborty et al. 2020, Khadka et al. 2020). Moreover, the use of Ca-based stabilizers, such as lime, as the primary stabilizer, increases the carbon footprint of the construction project, and hence researchers and engineers are continuously striving to identify alternate eco-friendly stabilizers that can be sustainably used for treating problematic soils (Zhang et al. 2015, Phummiphan et al. 2016, Samuel et al. 2021).

Geopolymer is one such sustainable soil stabilizer that has recently gained prominence due to its efficacy in reducing the swell-shrink characteristics and enhancing the strength and stiffness properties (Cristelo et al. 2011, Zhang et al. 2015, Samuel et al. 2019, Spagnoli et al. 2020). Geopolymers are inorganic cementitious products formed by alkali activation of alumino-silicate-rich materials, including metakaolin, fly ash, furnace slag, and red mud (Davidovits 2005, Duxson et al. 2007, Zhang et al. 2015, Khadka et al. 2020). Since most of these alumino-silicate rich materials are derived from waste products, the application of geopolymers reduces the carbon footprint and is considered an eco-friendly stabilizer (Spagnoli et al. 2020). Although researchers have reported that geopolymer stabilized soils exhibit comparable engineering properties as obtained with traditional stabilizers, most of the studies dealt with the treatment of low-sulfate soils. The limited number of studies on geopolymer treated high-sulfate soils focused on geopolymer treatment of sulfate-rich lean clays or considered geopolymer-lime combination for treating high-sulfate expansive soils (Zhang et al. 2015, Khadka et al. 2020, Yu et al. 2020). Furthermore, these studies primarily examined the swelling characteristics of the treated high-sulfate soils but did not investigate the impact of moisture intrusion on the strength properties of the geopolymer-treated soils.

An experimental program was designed and conducted to address the above-mentioned shortcomings by studying the swelling and post-soaking strength characteristics of lime-treated and geopolymer-treated expansive sulfate soils. Laboratory tests such as one-dimensional free

swell tests and unconfined compressive strength (UCS) tests, with and without moisture conditioning, were conducted to investigate the improvements in engineering properties of the geopolymer-treated soils over untreated and lime-treated soils. Additionally, field emission scanning electron microscope and electron dispersive x-ray spectroscopy (FESEM-EDXS) imaging of geopolymer-treated and lime-treated soil specimens was performed to study the microstructural changes after chemical treatments. The findings of this study highlight the scope of utilizing geopolymers as sustainable non-traditional stabilizers for treating sulfate-rich expansive soils. The following sections present the materials used in this study, metakaolin-based geopolymer (MKG) synthesis processes, engineering tests performed on the untreated and treated soils, and analyses and discussion of test results.

MATERIALS AND TESTING

Geomaterial. Natural soil was collected from a highway embankment construction site in North Texas. The basic characterization of the soil was performed as per ASTM or TxDOT standards. The natural soil was classified as high-plasticity clay (CH) as per Unified Soil Classification Systems (USCS), with a clay fraction of 57.5%, plasticity index of 33%, and free swell strain of 12.2% (maximum dry density (MDD) = 1.66 g/cm³ and optimum moisture content = 20%). Mineralogical studies indicated that the soil primarily consists of Kaolinite, Illite, Smectite, Silica, and a significant concentration of Calcite in its natural state.

Preparation of artificial sulfate-rich expansive soils. In this study, low-sulfate soil (LS) and high-sulfate soil (HS) were used for experimental investigations and comparative studies. Natural soil having a sulfate content of 336 ppm was adopted as LS (soluble sulfate concentration < 3,000 ppm). High-sulfate soil (HS) containing a sulfate content of 10,000 ppm was prepared by adding gypsum to natural soil. The prepared soils were treated with both MKG or lime and subsequently studied for engineering and microstructural properties.

Geopolymer synthesis. The basic ingredients for MKG synthesis consisted of KOH (18.2%), amorphous fumed silicon (IV) oxide (9.2%), metakaolin (MK) (37.6%), and deionized water (35.0%). First, a highly alkaline solution was prepared by mixing KOH crystals with the deionized water. Thereafter, the silica fumes were added to the alkaline solution to form an activating solution. A homogeneous solution was then prepared by uniformly mixing the MK with the activating solution to obtain the metakaolin-based geopolymer slurry with a target SiO₂/Al₂O₃ ratio. Although infinite MKG compositions are possible, MKG with SiO₂/Al₂O₃ = 3, water to solid ratio = 3, and K/Al = 1 imparted maximum unconfined compressive strength to treated specimens cured for 3 days and was therefore selected as the optimum geopolymer composition for this study.

Engineering tests. The optimum lime dosage of 6% was determined using the Eades and Grim pH test method (ASTM D6276-19). The dosages of MKG were selected as 8% and 30% of the dry weight of soils. The target dry densities and moisture contents for various soil groups treated with lime or MKG are shown in Table 1. All treated specimens were cured at room temperature of 23±2°C for 3 days and 14 days in a hermetically sealed chamber to ensure close to 100% relative humidity during the curing period.

Free swell tests were performed on untreated, lime-treated, and MKG-treated specimens. For repeatability, multiple specimens with the same diameter of 63.5 mm and a height of 25.4 mm were prepared by static compaction to reach the desired dry density and moisture content. Tests were performed under vertical stress of 1 kPa as per ASTM D4546-21. Dial gauge readings were recorded until there was no change in gauge readings for three consecutive days.

Table 1. Target dry densities and moisture contents for each specimen group.

Soil group	Stabilizer type and dosage	Specimen group	98% MDD (g/cm ³)	MC at 98% MDD on wet side (%)
Low-sulfate soil (LS)	6% Lime (6L)	6L-LS	1.47	30.0
	8% MK-based GP (8MKG)	8MKG-LS	1.45	28.1
	30% MK-based GP (30MKG)	30MKG-LS	1.56	24.6
High-sulfate soil (HS)	6% Lime (6L)	6L-HS	1.47	30.0
	8% MK-based GP (8MKG)	8MKG-HS	1.45	28.1
	30% MK-based GP (30MKG)	30MKG-HS	1.56	24.6

Note: MDD = Maximum Dry Density; MC = Moisture Content

For UCS tests, all cylindrical specimens were prepared using a Harvard miniature mold (33.3 mm in diameter and 67.3 mm in height). Triplicate specimens of each specimen group were prepared at 98% MDD and the corresponding moisture content on the wet side of the curve (Table 1). For soaked UCS tests and volumetric swell estimation, specimens were subjected to capillary soaking for 48 hours before tests. All UCS tests were performed on untreated, lime-treated, and MKG-treated specimens before and after capillary soaking at a strain rate of 1%/min per ASTM D2166-16.

A FESEM device with a maximum resolution of 1 nm was used to generate images of treated specimens for this study. Lime- and MKG-treated HS specimens cured for 14 days were subjected to FESEM-EDXS imaging to compare the microstructural changes and to detect the potential formation of new reaction products in the treated soils.

RESULTS AND DISCUSSION

Free swell tests were conducted on untreated, lime-treated, and MKG-treated specimens cured for 3 days and 14 days to study the effect of MKG in mitigating the swell behavior of the soil as compared to lime (Figure 1). The untreated soil experienced a free swell strain of 12.2% due to clay mineral-induced swelling, and a major part of the swell strain accumulated within one day of moisture exposure.

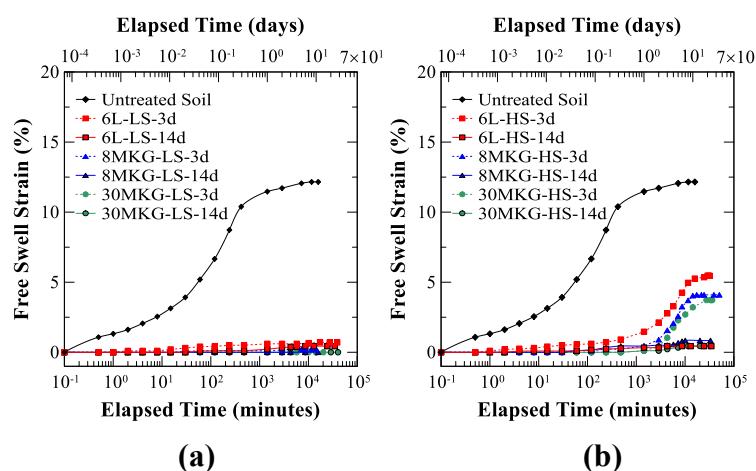


Figure 1. Free swell strain vs. elapsed time: (a) LS and (b) HS treated with lime or MKG.

For the LS soil, all the treatments were observed to provide a substantial reduction in free swell strain after three days of curing. The addition of lime to soils reduces the swelling potential through modification reactions and the formation of cementitious compounds for both curing periods. For the MKG-treated specimens, reduction in free swell can be partially attributed to the cation exchange between the clay minerals and potassium cations as well as the formation of the geopolymer-soil matrix over the curing period.

The lime-treated HS soil specimens did not swell immediately after moisture exposure. Instead, a substantial swelling was observed after one day of starting the swell test. This swelling characteristic is typical due to the nucleation and growth of ettringite in soils. During the initial soaking period of one day, the modification reactions and cementitious gels could resist the ettringite-induced heave. However, ettringite crystals that had nucleated and formed in the specimen gradually absorbed water during the test and exhibited ettringite-induced swelling (Figure 1b).

The MKG-treated soils cured for three days showed a similar trend to lime-treated specimens. The presence of an excess concentration of KOH during the early curing period and natural soil calcite probably shifted the reaction equilibrium towards the formation of K_2CO_3 and $Ca(OH)_2$. This secondary $Ca(OH)_2$ might have reacted and formed ettringite in the presence of sulfates and aluminates under a high pH environment. Consequently, for both MKG treatment dosages, an S-shaped curve similar to lime treatment was observed when subjected to the free swell test. However, the treated soil matrix was able to resist the ettringite-induced swelling when cured for a longer time due to the completion of the geopolymerization process and the formation of a strong geopolymer-soil network.

Table 2 shows the average unsoaked and soaked UCS of the untreated and treated soil groups with different curing periods. Unsoaked and soaked UCS values of the untreated soil were observed as 144.9 kPa and 13.0 kPa, respectively. All chemical treatments significantly improved the strength as compared to untreated soil for all curing periods. This could be attributed to several factors, including modification reactions, pozzolanic reactions, or geopolymerization. However, the engineering properties of certain treated specimens degraded after exposure to moisture intrusion. Therefore, the durability aspect of all treated soil groups was analyzed using the percentage retained strength (S_R) and volumetric strain (V_S) parameters. S_R is the ratio of the soaked strength to the unsoaked strength, expressed as a percentage. V_S is the ratio of volume change of a specimen to its initial volume, expressed as a percentage. These volumes were calculated using the diameter and height of the UCS specimens before and after capillary soaking for 48 hours.

Table 2. Average UCS values of treated soils.

Curing period (days)	6L-LS		6L-HS		8MKG-LS		8MKG-HS		30MKG-LS		30MKG-HS	
	US	S	US	S	US	S	US	S	US	S	US	S
3	362.6	257.0	481.9	120.3	375.5	92.6	267.6	65.9	2254.4	1565.2	2071.3	1800.4
14	616.1	437.5	732.7	278.3	545.0	152.9	317.8	127.7	2569.5	1866.5	2498.9	1862.4

Note: US = Unsoaked UCS; S = Soaked UCS; All units are in Kilopascal (kPa)

Figure 2 represents the S_R and V_S values of treated-LS specimens for different curing periods. The performance of both lime and 30% MKG treated specimens was observed to be similar for the LS soil groups for both curing periods. The cementitious gels formed due to the addition of Ca-based stabilizers were responsible for such behavior after lime treatment. For 30% MKG

dosage, the development of a uniform matrix due to substantial geopolymerization could be attributed as a primary reason for the improvement in performance. 8% MKG dosage might have resulted in the formation of insufficient physical bonding in the treated soil matrix, and as compared to lime treatment, the absence of pozzolanic reactions could have resulted in the poor performance for these treated soil groups.

The S_R and V_S values for the treated HS specimens are shown in Figure 3. It could be noted that the lime treatment and lower MKG dosage were not able to provide significant improvement in S_R and V_S values. The nucleation and growth of ettringite crystals resulted in significant volumetric strains, an increase in void ratios, and a subsequent reduction in soil strength after moisture intrusion. For lower MKG dosage, the insufficient strength of the MKG-matrix was unable to resist ettringite-induced heave. 30% MKG was able to provide significant bonding and prevent possible moisture intrusion due to the coating of soil particles and binding them to form a strong matrix. These factors were primarily responsible for the increased resistance to moisture intrusion for the 30% MKG-treated soils.

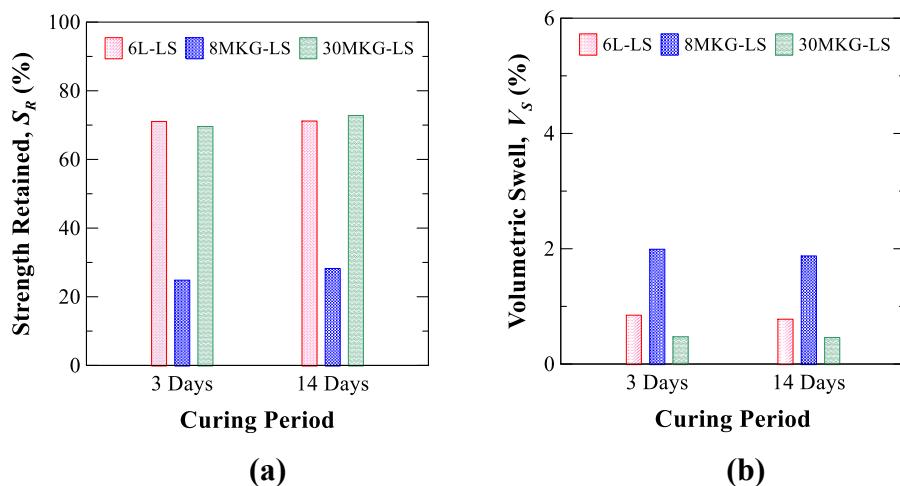


Figure 2. Low-sulfate soils (LS) treated with lime or MKG: (a) Percentage strength retained (S_R) after capillary soaking and (b) Volumetric swell (V_S) after capillary soaking.

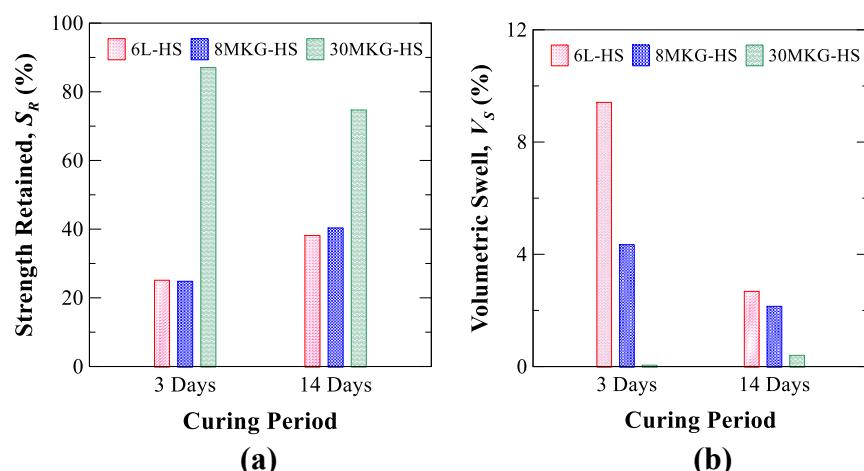


Figure 3. High-sulfate soils (HS) treated with lime or MKG: (a) Percentage strength retained (S_R) after capillary soaking and (b) Volumetric swell (V_S) after capillary soaking.

FESEM studies were performed to detect new chemical reaction products in 6L-HS and 30MKG-HS specimens cured for 14 days (Figure 4). Figure 4a shows tubular-shaped ettringite crystals formed in the 6L-HS specimens. The presence of ettringite is responsible for significant swelling, strength loss, and volumetric swell (Figures 1 and 3). 30MKG-HS specimens showed a uniform coating of a cementitious MKG matrix over the soil particles and no cracks were observed on the surface of the GP coatings (Figure 4b). This MKG matrix could be primarily responsible for enhancing the engineering properties of treated-HS specimens (Table 2). These bonds could also be responsible for providing resistance to expansion during moisture intrusion and subsequently reducing the swelling of the expansive soils (Figure 3).

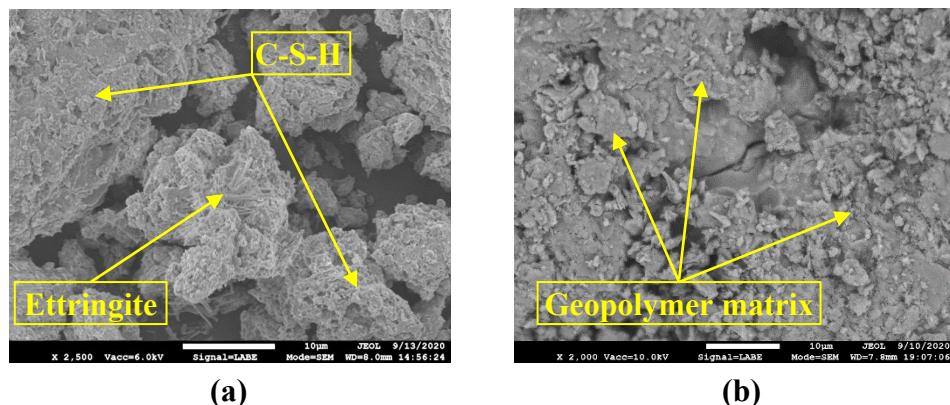


Figure 4. FESEM images: (a) 6L-HS showing the needle-shaped ettringite (2,500x magnification) and (b) 30MKG-HS showing the geopolymer matrix (2,000x magnification).

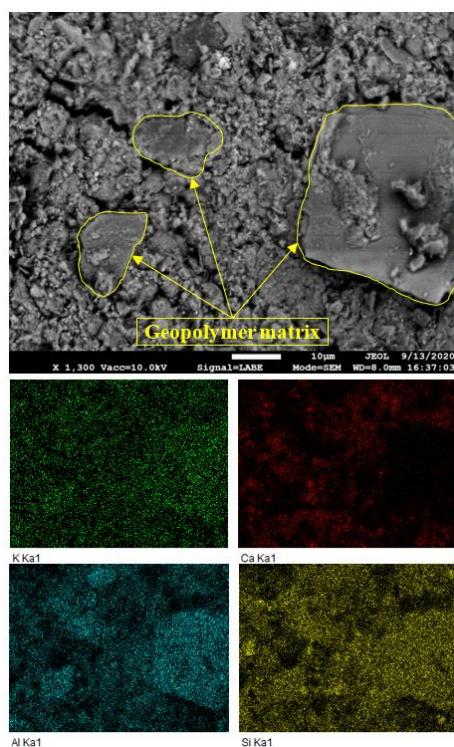


Figure 5. FESEM-EDXS elemental mapping of 30MKG-HS.

Figure 5 shows the EDXS mapping of the 30MKG-HS specimen cured for 14 days. It is observed that potassium from KOH is uniformly distributed on the soil particles and MKG matrix. The absence of calcium on the MKG matrix confirms its efficacy in reducing ettringite formation in sulfate-rich soils. Therefore, in clay minerals that contain a suitable concentration of calcium, silicon, and aluminum; an area indicating the absence of calcium after treatment can be used as a good indicator for predicting the formation of the MKG matrix.

CONCLUSIONS

This research study was conducted to evaluate the feasibility of utilizing metakaolin-based geopolymer for stabilizing expansive sulfate soils. Sulfate soils were treated with geopolymer, and the swelling and strength characteristics were studied at different stabilizer dosages and curing periods. The improvements in these engineering properties were compared with those obtained by using lime as the chemical stabilizer. FESEM-EDXS imaging was also performed to detect newly formed chemical reaction products in the treated soils. Some of the salient findings from this study are presented below.

- Lime treatment effectively reduced clay-mineral induced swelling in the expansive soils, irrespective of the sulfate levels. However, the high-sulfate soil exhibited ettringite-induced heaving, making lime treatment ineffective for sulfate-rich expansive soils. In contrast, geopolymer treatment was effective in reducing the swelling potential of the expansive soils at both sulfate levels. Prolonged curing and higher geopolymer dosage were especially beneficial in suppressing the swelling characteristics of the expansive sulfate soils.
- Both lime and geopolymer treatments were effective in enhancing the strength properties of the expansive soils. However, the lime-treated high-sulfate soil specimens experienced a significant strength loss and volumetric expansion when exposed to water. Geopolymer treatment of the same soil reduced the volumetric expansion and associated strength loss, especially at higher dosages.
- FESEM imaging showed ettringite crystals in the lime-treated specimens that were responsible for the swelling and strength reductions when exposed to water. Ettringite crystals were not detected in the geopolymer treated specimens. Instead, geopolymer was observed to coat the soil particles and bind them together. This physical binding effect of geopolymer reduced the swelling potential and enhanced the strength properties of the soil after treatment.
- FESEM-EDXS mapping showed the absence of calcium on the geopolymer matrix. Potassium, the main source of geopolymer synthesis, was distributed throughout the MKG-treated soils. These facts suggest that treated soils have a strong potential to form a uniform geopolymer matrix, and consequently reducing the possibility of ettringite formation.

Future studies are necessary to understand the potential reaction mechanisms involved during the process of geopolymerization and its interaction with high-sulfate soil. However, the preliminary studies indicate that this eco-friendly stabilizer has the potential to be used as a sustainable alternative stabilizer for treating high-sulfate soils.

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